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Characterization of Aluminum Oxide Films Deposited on Al_2O_3-TiC by RF Diode Sputtering

H. Panitchakan[a*] and P. Limsuwan[a,b]

[a]Department of Physics, King Mongkut's University of Technology Thonburi, Bangkok, 10140, Thailand
[b]Thailand Center of Excellence in Physics, CHE, Ministry of Education, Bangkok 10400, Thailand

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Abstract

In magnetic head recording storage, Al_2O_3 film was deposited onto Al_2O_3-TiC substrate, as insulating and protection layers, using RF diode sputtering. Pure Al_2O_3 (99.5%) with a dimension of 43.2 cm ×43.2 cm × 2.0 cm was used as a sputtering target. The base pressure for all depositions was 1×10^{-6} m Torr and the deposition time was 12.2 min. The target sputtering power and substrate bias voltage were varied from 4 to 8 kW and 80 to 180 V, respectively. The surface morphology of Al_2O_3 films was investigated using atomic force microscopy (AFM) and scanning electron microscopy (SEM). It was found that the roughness of deposited Al_2O_3 films depends on sputtering power. SEM cross-sectional image clearly showed good adhesion between substrate and Al_2O_3 film. The mechanical properties, including elastic modulus and hardness, of Al_2O_3 films were performed on nanoindentator. The results showed that both elastic and hardness of Al_2O_3 films increased with the increase of target sputtering power.

Keywords: Al_2O_3 film; Al_2O_3-TiC substrate; RF diode sputtering

1. Introduction

Aluminum oxide (Al_2O_3) or traditional name "alumina" is well known material used in variety applications in microelectronic, optical application, protection layer and magnetic head recording. Because of its desirable both mechanical and insulating properties [1]. In magnetic recording, Al_2O_3 film is used as undercoating layer to construct the devices on it and it is also used as over coating layer to seal off all construction devices at the final process including dielectric gap layer between the device layers [2-

* Corresponding author. Tel.: +662-872-5253; fax: +662-872-5254.
E-mail address: Hathai_Pa@hotmail.com.

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4]. Al_2O_3 films can be prepared with several methods such as atomic layer deposition, e-beam evaporation, filtered cathodic vacuum arc, reactive sputtering and non- reactive sputtering [5-11]. In particular, growth of Al_2O_3 film as insulator by non-reactive sputtering method is less complex process compared with reactive sputtering method. For Al_2O_3 films, obtained from non-reactive sputtering, the properties of Al_2O_3 films depend on the sputtering conditions, such as substrate bias voltage, target sputtering power and sputtering pressure. Thus, the aim of this work is to prepare Al_2O_3 films onto Al_2O_3-TiC substrate with Al_2O_3 target using RF diode sputtering, i.e. non-reactive sputtering. The sputtering conditions used for deposition of Al_2O_3 films are target sputtering power and substrate bias voltage. The deposited films were then characterized by atomic force microscopy (AFM).

2. Experimental

Al_2O_3 films were deposited by a commercial RF diode sputtering system (Comptech 2460). Pure Al_2O_3 of 99.5% purity was used as a sputtering target. The dimension of Al_2O_3 target is 43.2 cm in width, 43.2 cm in length and 2.0 cm thick. The substrate is Al_2O_3-TiC wafer with a round shape of 15.2 cm in diameter. The substrate was preliminary cleaned by high pressure spray of deionized water to remove small particles contamination and then they were cleaned in vacuum chamber with O_2 plasma to remove the native oxide layer. The target to substrate distance was 23.76 mm. The sputtering gas was Ar of 99.9% purity and He gas was used to cool the substrate. The chamber was evacuated to a base pressure of $2x10^{-6}$ Torr. Prior to the film deposition, the target surface and the substrates were cleaned by Ar^+ ions at a pressure of 2×10^{-2} Torr for 3 and 2 min, respectively. During cleaning, the target and the substrates were applied with the powers of 4 kW and 800 W, respectively.

Table 1. RF sputtering conditions for deposition of Al_2O_3 film in the first experiment

Parameters	Values
Target sputtering power (kW)	7
Substrate bias voltage (V)	80, 100,120,150,180
Operating pressure (mTorr)	24
Base pressure (mTorr)	$2x10^{-6}$
Deposition time(min)	12.2 min

Table 2. RF sputtering conditions for deposition of Al_2O_3film in the second experiment

Parameters	Values
Target sputtering power (kW)	4 ,5 ,6 ,7 ,8
Substrate bias voltage (V)	150
Operating pressure (m Torr)	24
Base pressure (mTorr)	$2x10^{-6}$
Deposition time(min)	12.2 min

Two experiments on the preparation of Al_2O_3 were conducted. In the first experiment, Al_2O_3 films were deposited at different substrate bias voltages from 80 to 180 V with an increasing step of 20 V. In the second experiment, Al_2O_3 films were deposited at various target sputtering powers from 4 to 8 kW with an increasing step of 1 kW. The depositing conditions of Al_2O_3 films are listed in Table1 and Table2.

The film thickness was measured by N&K analyzer (N&K Instrument). The surface morphology of Al_2O_3 film was analyzed by atomic force microscopy (AFM) [12]. The mechanical properties were carried out by adhesion test and film hardness test [13]. Adhesion between the film and substrate was performed by slicing the sample into a bar with a size of 1.24 mm × 45.72 mm × 0.25 mm using a high speed diamond blade at a cutting speed of 9000 rpm. The cross section of the bars were then analyzed by scanning electron microscopy [12]. The hardness test of deposited Al_2O_3 films was performed using a cube corner indenter of nanoindentator with a loading force of 150 μN and a convention depth of 10% of the film thickness.

3. Results and discussion

3.1. Deposition rate of Al_2O_3 film

The thickness of Al_2O_3 films obtained from two experiments corresponding to sputtering conditions as shown in Table 1 and Table 2 was measured by N&K analyzer and the results are given in Table 3 and Table 4. The deposition rate was then calculated and the results are also given in Table 3 and Table 4.

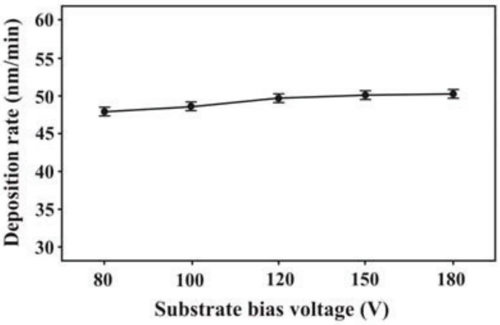
Table 3. Thickness and deposition rate of Al_2O_3 film for various substrate bias voltages

Substrate bias voltage (V)	Target sputtering power (kW)	Operating pressure (mTorr)	Deposition Time (min)	Thickness (nm)	Deposition rate (nm/min)
80	7	24	12.2	596	48.8
100	7	24	12.2	609	49.9
120	7	24	12.2	618	50.6
150	7	24	12.2	622	50.9
180	7	24	12.2	630	51.6

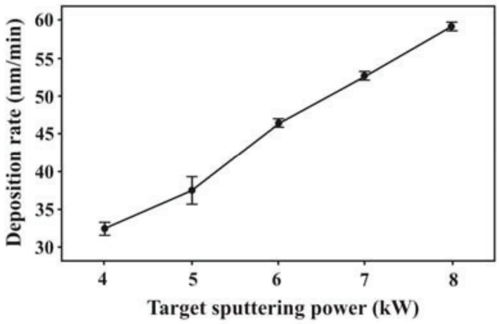
Table 4. Thickness and deposition rate of Al_2O_3 film for various target sputtering powers

Target sputtering power (kW)	Substrate bias voltage (V)	Operating pressure (mTorr)	Deposition Time (min)	Thickness (nm)	Deposition rate (nm/min)
4	150	24	12.2	407	33.4
5	150	24	12.2	464	38.0
6	150	24	12.2	565	46.3
7	150	24	12.2	636	53.0
8	150	24	12.2	709	58.1

The relation between the deposition rate and the substrate bias voltage is shown in Fig. 1 (a) and the relation between the deposition rate and the target sputtering power is shown in Fig. 1 (b)



(a)



(b)

Fig. 1. (a) Relation between the deposition rate and the substrate bias voltage and (b) relation between the deposition rate and the target sputtering power

It is clearly seen from Fig. 1(a) that the deposition rate is independent of the substrate bias voltage. On the other hand, the deposition rate almost linearly increases with the increase of the target sputtering power. Hence, it can be concluded that the target sputtering power is more influence on the deposition rate of Al_2O_3 film than the substrate bias voltage. The increase of the deposition rate with target sputtering power is due to the increase of positive ion or Ar^+ ion attacking on Al_2O_3 target and result in higher ionization to target. From this study, a high deposition rate of 0.96 nm /sec was obtained at a target sputtering power of 8 kW and a substrate bias voltage of 150 V.

3.2. Surface morphology of Al_2O_3 film

The surface morphology of Al_2O_3 film was investigated by an optical microscope with a magnification of 200X objective in order to determine the defects in the film and the film characteristic. Fig. 2 shows the photograph of Al_2O_3 film deposited at a target sputtering power of 7 kW and a substrate bias voltage of 150 V for 12.2 min. Fig. 2 indicates that the Al_2O_3 film is transparent and hence we can see the grains of Al_2O_3 –TiC substrate at the underneath of Al_2O_3 film. In the picture, Al_2O_3 grains are black and TiC grains are white. The defects in the film can also be observed as shown by a black area in the circle. The presence of the defects is due to the residual aluminum oxide contamination on the chamber, shutter and shielding fall down onto the substrate during deposition. Therefore, the chamber cleaning before the Al_2O_3 film deposition is required.

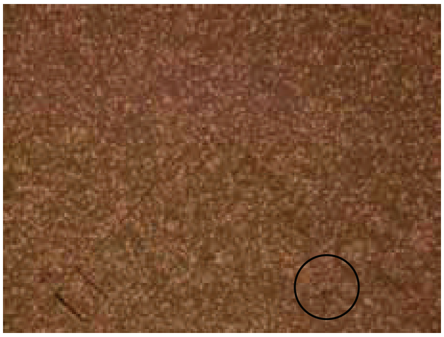


Fig. 2. Photograph of of 0.6 μm thick Al_2O_3 film deposited on Al_2O_3-TiC substrate with a magnification of 200X

Atomic force microcopy (AFM) was used to investigate the surface roughness of Al_2O_3 films. Figure 3 (a-d) shows AFM images of Al_2O_3 films prepared at a constant substrate bias voltage of 150 V and different target sputtering power of (a) 8 kW, (b) 7 kW, (c) 6 kW and (d) 5 kW. The roughness was measured by scanning the AFM tip pass through the center of the sample The scan size for all images was 5×5 μm^2. The RMS roughness and R_{max} are given in Table. 5. It can be seen that the roughness of Al_2O_3 film surface significantly increases with target sputtering power.

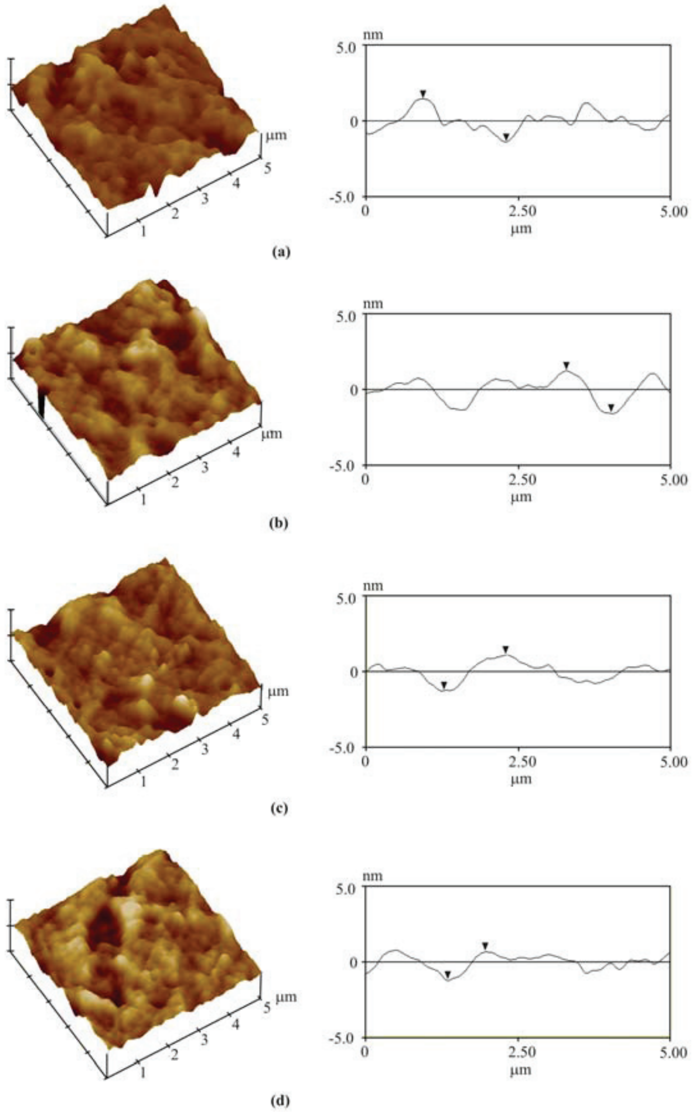


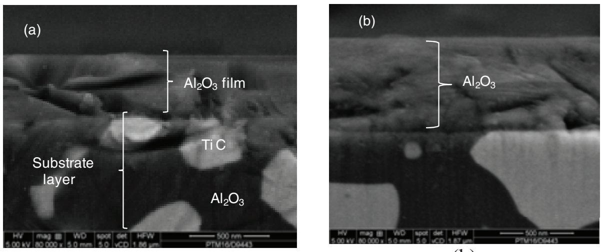
Fig. 3. 3D AFM image and cross section of Al_2O_3 films deposited at different target sputtering powers of (a) 8 kW, (b) 7 kW, (c) 6 kW and (d) 5 kW

The values of roughness shown in Table 5 are two characteristics surface roughness (i) the root –mean –square (RMS) roughness and (ii) the difference between the highest and lowest points in the scan range (Rmax). The plots between the roughness and the target sputtering power is shown in Fig. 4. It is seen that the roughness increased as the target sputtering power was increased.

3.3. Mechanical properties

3.3.1. Adhesion property

The Al_2O_3 films deposited at different target sputtering powers of 5, 6, 7 and 8 kW were used for adhesion test. Each sample was sliced into a bar of dimension 1.24 mm × 45.72 min × 0.25 min. The cutting edge of each sample was investigated by SEM. Fig. 6 shows typical cross-section images of SEM micrograph for Al_2O_3 films deposited at (a) 6 kW and (b) 8 kW. It was found that all deposited Al_2O_3 films show no delaminating from Al_2O_3-TiC substrate. Furthermore, Fig. 5 confirms that the Al_2O_3 film deposited at 8 kW is thicker than that of 6 kW.



(a) (b)

Fig. 5. SEM micrograph of Al_2O_3 film deposited at target sputtering powers of (a) 6 kW and (b) 8 kW

Table 6. Hardness and elastic modulus of Al_2O_3 films deposited at various target sputtering powers

sputtering power (kW)	Substrate bias voltage (V)	Elastic Modulus (GPa)	Hardness (GPa)
5	150	27.6	6.6
6	150	50.3	7.2
7	150	61.9	7.5
8	150	62.8	7.6

3.3.2. Hardness property

The hardness of deposited Al_2O_3 films obtained by nanoindentator with a cube corner of curvature radius of 150 nm and a loading force of 150 μN is given in Table 6. The plots between the hardness with the target sputtering power and between the elastic modulus with the target sputtering power are shown in Fig. 6. It is seen that the roughness increased as the target sputtering power was increased.

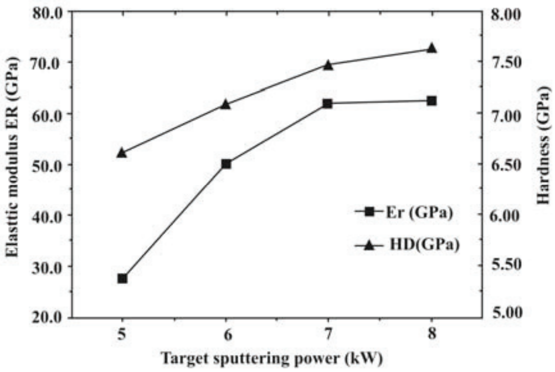


Fig. 6. The plots of elastic modulus and hardness of Al_2O_3 film with target sputtering power

It is seen from Table 6 that the hardness of Al_2O_3 film obtained in this work is in the range of 6.62 to 7.62 GPa. Furthermore, the hardness and the elastic modulus increased with the increase of target sputtering power from 5 kW to 8 kW while elastic modulus (Er) is obviously increase from between 27.6 GPa to 62.8 GPa.

4. Conclusion

In this work, Al_2O_3 films were deposited onto Al_2O_3-TiC substrate using RF diode sputtering. The target sputtering power and the substrate bias voltage were varied from 4 to 8 kW and 80 to 180 V, respectively. It was found that the deposition rate of Al_2O_3 films depended only on the target sputtering power. The maximum deposition rate of 0.96 nm/sec was obtained for a target sputtering power of 8 kW and a substrate bias voltage of 150 V. Furthermore, the RMS roughness and hardness of the films slightly increased with the increase of target sputtering power.

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